

## Startup of Savannah River's Defense Waste Processing Facility to Produce Radioactive Glass

by

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**Startup of Savannah River's Defense Waste Processing Facility  
To Produce Radioactive Glass**

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## **ABSTRACT**

The Savannah River Site (SRS) began production of radioactive glass in the Defense Waste Process Facility (DWPF) in 1996 following an extensive test program discussed earlier (S. J. Marra, et. al., THE DWPF: RESULTS OF FULL SCALE QUALIFICATION RUNS LEADING TO RADIOACTIVE OPERATIONS, Proceedings - Waste Management '96, Tucson, AZ). Currently DWPF is operating in a 'sludge-only' mode to produce radioactive glass consisting of washed high-level waste sludge and glass frit. Future operations will produce radioactive glass consisting of washed high-level waste sludge, precipitated cesium, and glass frit. This paper provides an update of the processing activities to date, operational problems encountered since entering radioactive operations, and the programs underway to solve them.

## **INTRODUCTION**

In March, 1996, the DWPF began the task of vitrifying approximately 130 million liters of high-level radioactive waste into borosilicate glass. This waste which is stored in carbon steel underground tanks is being pretreated, melted and poured into stainless steel canisters for eventual disposal in a geologic repository.

Prior to beginning radioactive operations the DWPF completed a Waste Qualification Run (WQR) of fifty-five canisters using various compositions of simulated waste. The extensive characterization of the glass and canistered waste form demonstrated that the DWPF can comply with the Department of Energy Office of Environmental Management's Waste Acceptance Product Specifications (WAPS).<sup>1,2,3</sup>

The DWPF facility is now processing radioactive waste. Through July, 1997, DWPF has produced twenty-seven batches of radioactive feed; one hundred eighty canisters have been filled. Characterization of the canistered waste form in accordance with the program described in the Waste Qualification Report has continued to demonstrate compliance with the WAPS.

The DWPF is a technically complex facility and it was difficult to transition to radioactive operations. Glass pouring has been impacted on several occasions by unstable flow from the melter into the canisters ("wicking"). The engineering process utilized to troubleshoot this issue is discussed as well as the technical evaluation of the data collected during wicking events. Enhancements to provide more measurement rangeability, faster control loop scan times and

improved algorithms and control parameters have been implemented. Ultimately, a modification of the melter pour spout was necessary to solve the wicking problems encountered.

## PROCESS DESCRIPTION

The radioactive waste in the Savannah River Site (SRS) Tanks Farms is in two forms; a water soluble salt solution and saltcake, and an insoluble sludge of metal hydroxides and oxides. Therefore, DWPF receives two separate feed streams.

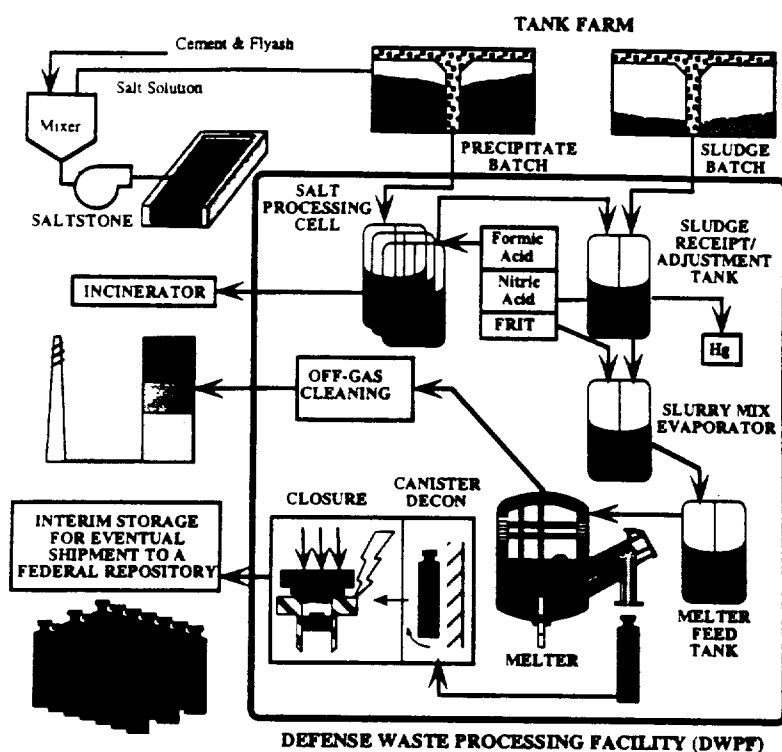


Figure 1. Immobilization of Savannah River Site Waste.

The salt solution and saltcake are decontaminated for disposal as low-level radioactive waste by the addition of sodium tetraphenylborate to precipitate the soluble salts of cesium and non-radioactive potassium and the addition of sodium titanate to adsorb residual strontium and plutonium. The resulting slurry is filtered and the decontaminated filtrate is blended with cement, slag and flyash for disposal at SRS as a low-level radioactive waste ("saltstone"). A sufficient quantity of washed concentrated precipitate is expected to be available for transfer to the DWPF

for immobilization in early 2000. Precipitate from the In-Tank Precipitation facility (ITP) will be transferred to the Late Wash (LW) facility for nitrite removal. The washed precipitate will then be transferred to the Low Point Pump Pit (LPPP) and hence to the Precipitate Reactor (PR) in the Salt Process Cell (SPC) for processing.

The precipitate is processed in the SPC to remove most of the organic material. The tetraphenylborate compounds comprising the precipitate react in the presence of formic acid and copper (II) catalyst. The products of this reaction are aromatic organic compounds (primarily benzene) and an aqueous phase known as Precipitate Hydrolysis Aqueous (PHA). The PHA contains the cesium formate, other soluble formate salts, boric acid, and excess formic acid. Due to problems encountered during ITP startup, DWPF is currently operating on a sludge only flowsheet. Initial operations involved production of a simulated PHA for use in the SRAT. This simulated PHA consisted of a solution of dilute formic acid and copper (II) catalyst in the form of copper nitrate. Later work showed that the copper nitrate was not need to produce acceptable glass so now concentrated formic acid is introduced directly into the SRAT.

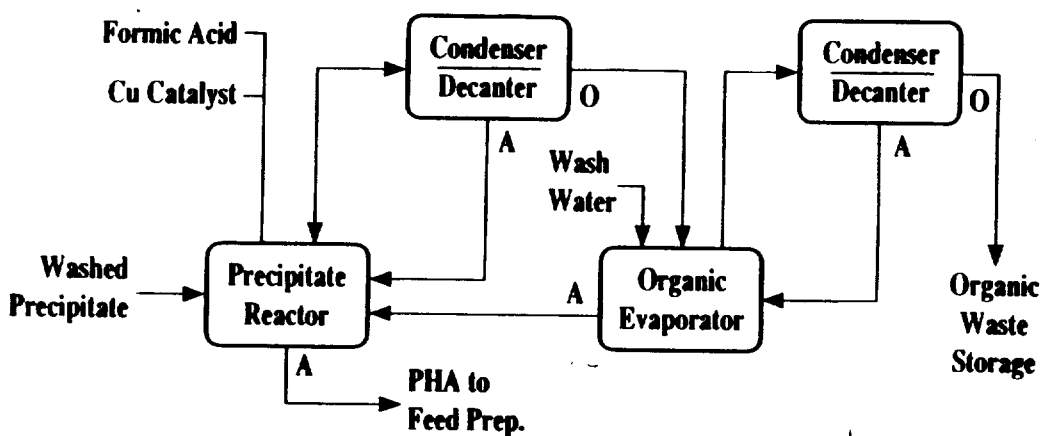


Figure 2. Precipitate Hydrolysis

The sludge waste is washed in the Extended Sludge Processing facility (ESP) to remove soluble salts. If necessary, insoluble aluminum is removed through high temperature caustic dissolution. Sludge slurry transfers began in March, 1996. Sludge slurry is transferred from ESP to the LPPP and hence to the Sludge Receipt and Adjustment Tank (SRAT) in the Chemical Process Cell (CPC) for processing. The sludge is transferred directly into the SRAT and then neutralized with nitric acid. Formic acid is then added to the sludge (at boiling). After the formic acid and sludge

are blended and processed in the SRAT, the product is transferred to the SME where a borosilicate glass frit is added and the slurry is concentrated to produce melter feed. The amount of sludge, nitric acid, and formic acid to be blended in the SRAT and the amount of SRAT product and frit to be blended in the SME is determined by the desired glass composition. This region of desired composition is determined by a series of glass property models and statistical algorithms. Any point within the acceptable region can be selected the target for a particular batch. Following acceptable sample results based on predicted glass properties the slurry is transferred to the Melter Feed Tank (MFT), and used as feed for the melter.

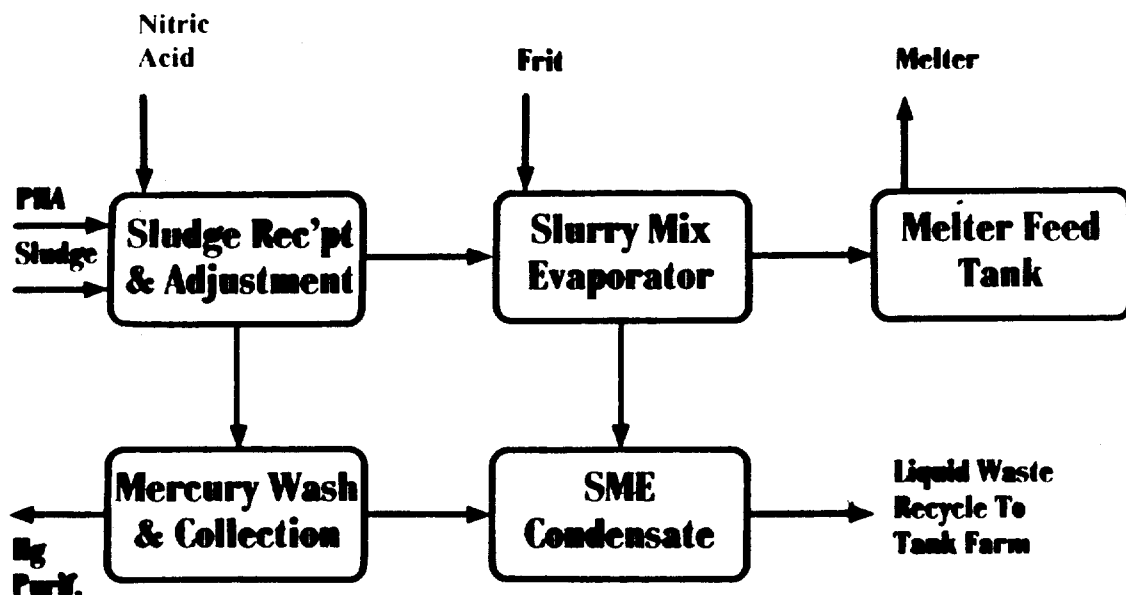


Figure 3. Melter Feed Preparation

The SME is the hold point in the process. The analysis of samples from the SME are used by the DWPF engineers to determine the acceptability of the batch versus the WAPS. The WAPS are divided into five sections: wasteform (borosilicate glass), canister, canistered waste form, quality assurance, and documentation. The most important of the glass specifications is the product consistency specification which states that the DWPF must control its process so that the glass produced is more durable than the DWPF Environmental Assessment Glass<sup>6</sup> as measured by the Product Consistency Test (PCT). Crushed glass is leached in distilled water and the concentration of boron, lithium, and sodium in the leachate are a direct measure of the durability. Durability is estimated from the composition of the SME slurry by summing the free energy of

hydration for each reactant. This approach has extensive empirical confirmation. Acceptance is based on this prediction. No material is allowed to be transferred from the SME to the MFT until it has been determined to be acceptable. A glass pour stream sample is taken occasionally during filling of a canister to confirm that the glass durability (as determined by the PCT<sup>7</sup>) is acceptable (see below).

Once the melter feed material in the SME is determined to be acceptable, it is transferred to the Melter Feed Tank (MFT) and then fed to a joule heated melter with two pairs of diametrically opposed electrodes. The feed slurry is introduced from the top of the melter and forms a crust, or cold cap, on the surface of the melt pool as the water is evaporated and removed via the off-gas system. The cold cap melts from the bottom and forms a borosilicate glass matrix. The nominal

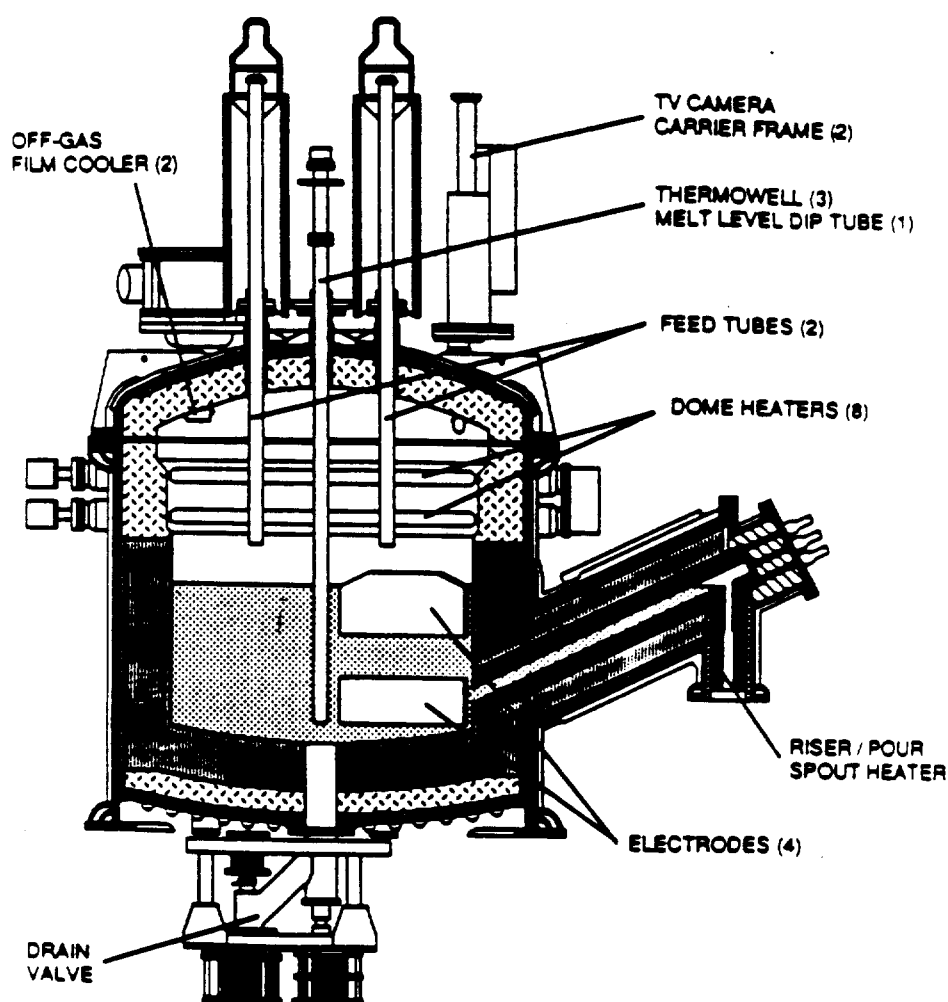


Figure 5. DWPF Glass Melter



glass pool temperature is 1130 °C. Since the residence time in the DWPF melter is long (~60 hours), the convective mixing is sufficient to ensure a homogeneous mixture.<sup>3</sup> The glass is removed from an opening near the bottom through a riser and pour spout. A vacuum is drawn on the pour spout to pour the glass into stainless steel canisters.

After a canister is filled, a temporary seal is installed to prevent free liquid from entering the canister during the decontamination process. The canister surface is decontaminated by blasting a frit/water slurry against the canister with compressed air. The frit slurry from decontamination is used in the next SME batch as part of the required frit addition. The canister is then welded closed and transferred to the Glass Waste Storage Building (GWSB) by the Shielded Canister Transporter (SCT).

## **INITIAL NON-RADIOACTIVE OPERATIONS**

Prior to radioactive operations several different campaigns were performed to validate the glass making process. Cold Chemical Runs were performed to demonstrate the system chemistry using a blended simulant to represent a composite of SRS tank farm waste. Waste Qualification Runs were performed using simulated feed types representing the extremes of the compositional envelope to verify the model developed to predict glass quality based on slurry chemistry in the SME.<sup>3</sup> Proficiency Runs were performed using simulated feed similar to that expected for initial radioactive runs to develop operational experience while waiting for Department of Energy (DOE) approval to enter full radioactive operations.

## **RADIOACTIVE OPERATIONS**

DWPF received approval from DOE to begin radioactive operations in March, 1996, and the first transfer from ESP to the LPPP of approximately 800 gallons of sludge into a receiving tank containing 3500 gallons of simulated sludge was made. This material was subsequently transferred to the SRAT for nitric acid addition and then to the SME for frit addition and acceptance. Through July, 1997, twenty-seven feed preparation cycles have been processed and one hundred eighty canisters filled.

During the first year of operations, problems like melter wicking, and failure of level and density sensing equipment in the process vessels, have been corrected or mitigated.

## **LEVEL GAUGES**

In vessels containing radioactive slurry, level and density measurements are determined using four Holledge™ sensors located at different levels in the vessel. Holledge™ sensors are used instead of standard bubbler because of the need to know level and density using an instrument that will not become plugged when used in sludge. Testing determined that a standard bubbler becomes plugged within 24 to 48 hours of operation in radioactive sludge. This due to sludge drying on the wall of the bubbler at the air to sludge interface and building up until it has bridged over the cross section of the pipe. The Holledge™ design uses a membrane sensor to measure the pressure exerted by the height of fluid covering the sensor. The membrane eliminates the air to fluid interface preventing the drying action that plugs a standard bubbler.

Performance of the Holledge™ level gauges in most of the process tanks has been a success. However, in the SME two level gauges failed in the first nine months of operation, with the second failing after only two months of operation. The mode of failure has not been determined, but both level gauges had an initial sensor fail with the remaining sensors failing in rapid succession. Alternate designs are under evaluation to determine if a better technology is available for this application.

## **MELTER POUR SPOUT WICKING**

The most serious operational problem to date has been wicking of glass in the melter pour spout. Glass pouring occurs by pulling a vacuum relative to the melter vapor space on the canister and pour spout. The glass level increases in the riser and overflows into the pour spout. The glass then travels vertically down the side wall approximately 38 cm where the wall of the pour spout is cut back to form a sharp 'knife edge' to allow the glass to disengage. The glass then freefalls a distance of about 60 cm through an unheated bellows and into the neck of the canister and up to 300 cm more to the bottom of the canister. Any glass which comes into contact with the unheated bellows has a tendency to adhere to the stainless steel surfaces and will build-up sufficiently to completely block the flow of glass from the pour spout to the canister. Wicking is the term used to describe contact of the glass stream with the unheated bellows or lower section of the pour spout below the knife edge.

The original pour spout design used in the test facility at SRS consisted of a two inch diameter downcomer. Glass was supposed to travel down the wall of this downcomer and disengage at the bottom of the pour spout and fall into the canister. This design did not work. The length of the

downcomer was such that the liquid glass would cool below the pour temperature and begin to solidify in the pour spout. Consequently, the pour spout was constantly plugging. This pour spout was modified by boring out a portion of the wall producing a 'knife edge' such that the glass stream would disengage at the knife edge while still above its pour temperature. During design of the production melter this knife edge was moved further up the pour spout to a point where the glass stream would disengage near its maximum temperature. This had the unfortunate consequence of requiring the glass stream to fall almost completely vertical since even a slight deviation would cause the glass stream to impact the colder pour spout wall. The pour spout was then modified to create a second knife edge to give the glass stream a second disengagement point should it be required. This had limited success however, experience showed that once the glass stream wicked off the first knife edge it tended to remain in contact with the pour spout wall surface. Several strategies were employed while searching for a solution to this problem.

Melter pouring is primarily controlled through the melter vapor space pressure controller and the melter pour spout to vapor space dP controller. The pour spout vacuum jet pump is a standard eductor unit operation and is the device used to generate vacuum on the pour spout section of the melter. The spout jet pump has a speed controller tied to it and runs at either low speed or high speed in which high speed is used during pouring. A plan of action was developed to carry out a series of open and closed loop evaluations of the two pressure control loops. This plan of action was executed by exercising the two loops in an open loop configuration over a number of operating boundaries. From the data collected the loops were determined to be coupled via a control air service line and as a result the tuning parameters were recalculated to de-couple the two loops. Details of this process may be found in reference 4. Unfortunately, this effort only provided a modest improvement in the wicking problem.

At the same time a tele-robotic manipulator (TRM) was designed, fabricated, and installed to assist with cleaning the pour spout.<sup>5</sup> The TRM is computer command controlled and has the capability to be pre-programmed to perform some evolutions automatically. It uses stationary and rotary tools (powered by the TRM) to clean the pour spout. It also has a camera mounted at the end of its arm to allow viewing of the pour spout. Improvement in pouring was noticed following each use of the TRM. However, this improvement was only temporary and multiple cleanings were sometimes required to pour one canister.

None of these solutions completely prevented wicking. In an effort to improve the pouring characteristics of the glass stream, a pour spout insert was developed to alter the pour spout geometry. The pour spout insert lowers the first knife edge. This allows for a greater deviation

from vertical as the glass stream falls from the pour spout to the canister. Following an extensive design and testing phase, the insert was installed in May, 1997. Since that time there have been no wicking problems.

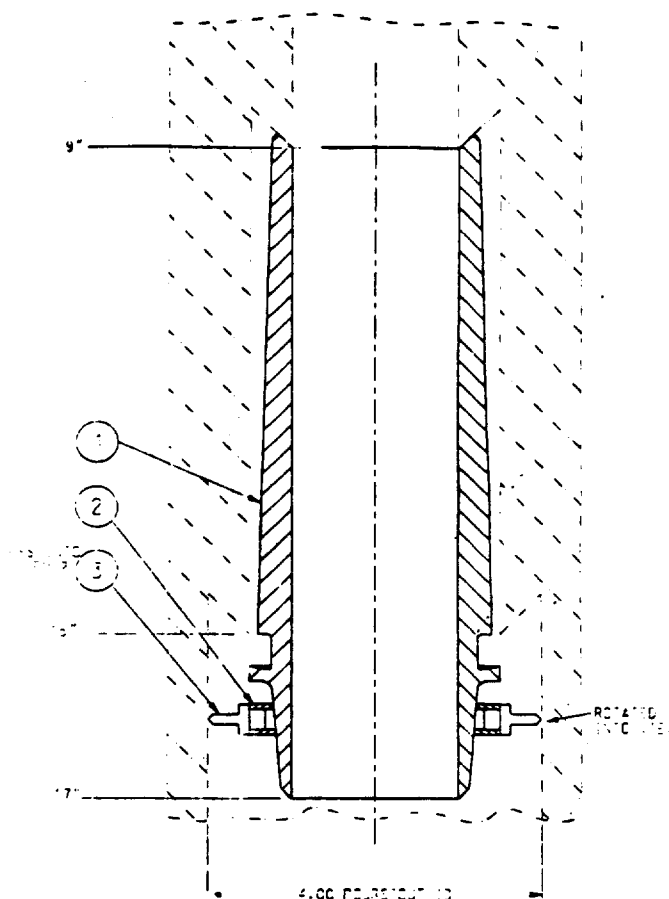


Figure 5. DPWF Melter Insert Subassembly

## CONCLUSION

In summary, the DWPF has begun the task of vitrifying radioactive waste into a borosilicate glass. Through July, 1997, DWPF has produced one hundred eighty canistered waste forms which meet the USDOE requirements for long term disposal in a geological repository.

Problem areas were identified and corrected in the melter pouring control system. A pour spout insert was successfully developed and installed to eliminate pour stream wicking events. Use of this insert will be incorporated in future melters at DWPF.

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3. S. L. MARRA, D. E. SNYDER, H. H. ELDER and J. E. OCCHIPINTI, "The DWPF: Results of Full Scale Qualification Runs Leading to Radioactive Operations", **Proceedings - Waste Management '96, CD-ROM Session 8-2**.
4. J. T. Carter, K. J. Rueter, J. W. Ray, and O. Hodoh, "Defense Waste Processing Facility Radioactive Operations - Part II Glass Making", **Proceedings - Waste Management '97**.
5. D. B. Little, J. T. Gee, and W. M. Barnes, "Defense Waste Processing Facility Radioactive Operations - Part I Operating Experience", **Proceedings - Waste Management '97**.
6. U. S. Department of Energy, **Environmental Assessment - Waste Form Selection for SRP High-Level Waste**, USDOE Document DOE/ES-0179, Washington, DC (1982).
7. C. M. JANTZEN, N. E. BIBLER, D. C. BEAM, W. G. RAMSEY, and B. J. WATERS, " Nuclear Waste Product Consistency Test (PCT) - Version 7," USDOE report WSRC-TR-90-539 rev 3, Savannah River Technology Center, Aiken SC (1994).

# Westinghouse Savannah River Company Document Approval Sheet

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Organization Code W8160	Organization (No Abbreviations) High Level Waste Management Division			
Other Authors Herbert H. Elder			Deadline Date for Approval 8/28/97 RCD 9/6/97	
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Author's Name: W. M. Bennett

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Department: HLWM

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Meeting Date: 9/8/97-9/12/97

References ☒ Approved for Release/Publicly Available ☐ Routed Concurrently  
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2. Award/Contract Nos.

DE-AC09-96SR18500

3. Title

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DOE-SR

5. B&R Code(s)

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7. CRADA Nos.

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9. Information Product Filename

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b. Conference title (No abbreviations) American Chemical Society Las Vegas National Meeting

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**E. Abstract**

The Savannah River Site (SRS) began production of radioactive glass in the Defense Waste Process Facility (DWPF) in 1996 following an extensive test program discussed earlier (S. J. Marra, et. al., THE DWPF: RESULTS OF FULL SCALE QUALIFICATION RUNS LEADING TO RADIOACTIVE OPERATIONS, Proceedings - Waste Management '96, Tucson, AZ). Currently DWPF is operating in a 'sludge-only' mode to produce radioactive glass consisting of washed high-level waste sludge and glass frit. Future operations will produce radioactive glass consisting of washed high-level waste sludge, precipitated cesium, and glass frit. This paper provides an update of the processing activities to date, operational problems encountered since entering radioactive operations, and the programs underway to solve them.

**F. Subject Terms** DWPF

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